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IS 14982 (2001): Anti-Striping Agents (Amine Type) [PCD 6: Bitumen Tar and their Products]

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एन्टी-स्ट्रिपिंग पदार्थ (एमीन टाइप) — विशिष्टि

Indian Standard

ANTI-STRIPPING AGENTS (AMINE TYPE) — SPECIFICATION

ICS 75.140

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

November 2001

Price Group 4

Bitumen,Tar and their Products Sectional Committee, PCD 6

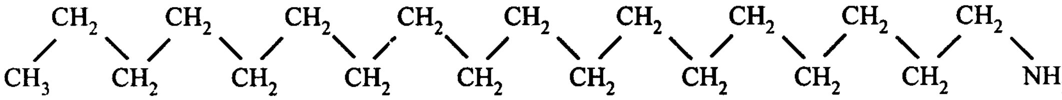
FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Bitumen, Tar and their Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

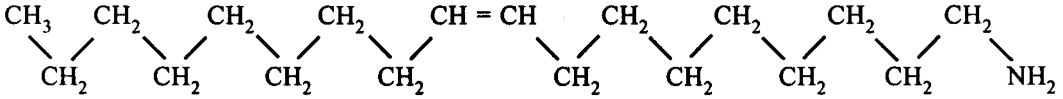
The adhesion between bitumen and aggregate is an important factor for the stability of bituminous pavements. Very often the cause of damage in road surfacings can be attributed to adhesion failure or stripping of bitumen film from aggregates.

Water during certain conditions has the ability to strip bitumen from aggregate and once this process starts it is accelerated by traffic. Most of the aggregates are more easily wetted by water than by bitumen.

Anti-stripping agents are used the world-over, to overcome the stripping problem in bituminous pavements. Anti-stripping agents improve the bond between binder and aggregate. In the presence of anti-stripping agents, the nature of aggregate surface is changed and aggregate becomes more liophilic (oil loving). Most commonly used anti-stripping agents now-a-days are long chain organic amines of relatively high molecular weight. The fatty amines having long hydrocarbon chain are the most suitable anti-stripping agents for bitumen. They are called fatty amines, as they are produced from fatty acids. To the long fatty hydrocarbon chain an amino group-NH_2 is attached. The structure of a saturated amine octadecylamine is shown as under:


CH_2 CH_2 CH_2 CH_2 CH_2 CH_2 CH_2 CH_2 CH_2
CH_3 CH_2 CH_2 CH_2 CH_2 CH_2 CH_2 CH_2 CH_2 NH_2

The physical properties of fatty acid amines depend on the length and nature of the hydrocarbon chain. The shorter the chain, the lower the melting point. Unsaturated hydrocarbon chains give lower melting point than saturated ones. For example, oleyeamine (unsaturated) is liquid at a temperature of 25°C. The typical chemical formula for oleyeamine is as given below:


CH_3 CH_2 CH_2 CH_2 $CH = CH$ CH_2 CH_2 CH_2 CH_2
CH_2 CH_2 CH_2 CH_2 CH_2 CH_2 CH_2 CH_2 NH_2

The hydrogenated tallow (saturated) has a melting point of about 50°C. The chemical properties of fatty acid amines can be varied by changing the number of amine groups and their positions in the molecule. Stearyl di-amine is an example of fatty acid amine which is often used as anti-stripping agent. It contains both primary and secondary amine groups.

Each molecule of these cationic fatty acid amines has a little 'head' which attaches itself with incredible force to the stone aggregate surface irrespective of the presence of water on the aggregate surface; and an elongated 'body' which holds on equally strongly to the binder, thus ensuring that the solid is coated with a regular layer of binder. The balance between the length of the hydrocarbon chain and the number of amine groups has therefore great influences on the adhesive power of doped bitumen. Short hydrocarbon chains are less soluble in bitumen than longer ones and their bond to the bitumen is weaker. Optimum performance is obtained with 14-18 carbon atom chain amines, with one or especially two amine groups, one of which is primary amine group.

This standard has been prepared for the guidance of the users and manufacturers of the anti-stripping agents of fatty amine type.

The Composition of the Committee responsible for formulation of this standard is given in Annex F.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

ANTI-STRIPPING AGENTS (AMINE TYPE) — SPECIFICATION

1 SCOPE

1.1 This standard prescribes the physical, chemical and performance requirements of anti-stripping agents (fatty acid amine type) used in bituminous materials for use in road pavements.

1.2 This standard also provides guidance with respect to applications, storage, handling, safety precautions, and additive dosage of anti-stripping agents.

2 REFERENCES

The following standards contain provisions which, through reference in the text, constitute provisions of this standard. At the time of publication the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards listed below:

IS No.	*Title*
73 : 1992	Paving bitumen (*second revision*)
334 : 1982	Glossary of terms relating to bitumen and tar (*second revision*)
1202 : 1978	Methods of testing tar and bituminous materials — Determination of specific gravity (*first revision*)
1209 : 1978	Methods for testing tar and bituminous materials — Determination of flash point and fire point (*first revision*)
1211 : 1978	Methods for testing tar and bituminous materials — Determination of water content (Dean and Stark method) (*first revision*)
1448 [P : 10] : 1970	Methods of test for petroleum and its products: P:10 Cloud point and pour point (*first revision*)
1448 [P : 86] : 1977	Methods of test for petroleum and its products: P : 86 Determination of total base number by potentiometric perchloric acid titration method
5194 : 1969	Method for determination nitrogen— Kjeldahl method
6241 : 1971	Method of test for determination of stripping value of road aggregates

3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 334 and the following shall apply.

3.1 Stripping

Stripping is the breaking of the adhesive bond between the aggregates and the bitumen or tar. In one way or the other water gets in between the bitumen film and the aggregate surface and because the aggregate surface has higher attraction for water than for bitumen the adhesive bond is broken.

3.2 Active Adhesive

If an anti-stripping agent is added to the bitumen, even wet aggregate surface may be coated. This is defined as active adhesive.

3.3 Passive Adhesive

If bitumen does not have the power to coat wet aggregate, but has coated dry aggregate and the contact angle between water, aggregate and bitumen does not change when immersed in water, it is defined as passive adhesive.

4 MATERIALS

Any suitable long chain fatty acid amine of bitumen, which is effective to prevent the stripping with any aggregate, may be used. It shall not have any adverse effect on bitumen properties (*see* IS 73) when incorporated in optimized dosage.

5 REQUIREMENTS

5.1 Anti-stripping agent either liquid or powder shall be homogeneous materials having tolerable odour.

5.2 The physical, chemical and performance requirements of the anti-stripping agents shall comply with the requirements specified in Table 1.

6 SAMPLING

6.1 For the purpose of testing, the sampling procedure from drums, barrels or tank shall be as follows:

> The content of drum or barrel from which the sample is to be drawn shall be warmed up to 45-55°C, if required, and thoroughly mixed by rolling to and fro on the ground for a period of 5 to 10 min. Where practicable, the content of that tank shall be agitated by forced circulation or air before sampling.

Table 1 Requirements for Anti-Stripping Agents (Amine Type)

(*Clause* 5.2)

Sl No.	Characteristic	Requirement	Method of Test, Ref to
(1)	(2)	(3)	(4)
i)	Specific gravity at 27°C	0.85-0.95	IS 1202
ii)	Pour point °C, *Max*	42	IS 1448 [P : 10]
iii)	Flash point, °C (COC), *Min*	125	IS 1209
iv)	Water content, percent by volume, *Max*	1.0	IS 1211
v)	Solubility in high speed diesel oil in the ratio of 2:98 at ambient temperature	Complete	Annex A
vi)	Thermal stability at 163°C, 5 h	No appreciable change in efficacy	Annex B
vii)	Total base number mgKOH/g, percent by mass, *Min*	200	IS 1448 [P : 86]
viii)	Nitrogen content, percent by mass, *Min*	7.0	IS 5194
ix)	Under water coating test with 1 percent anti-stripping, agent (Active adhesion)		
	a) For sprayed work	Complete coating	Annex C
	b) For premix work	do	Annex D
x)	Stripping value with bitumen containing 1 percent of anti-stripping agent (Passive adhesion)	No stripping	IS 6241
xi)	Retained Marshall stability	90	Annex E

NOTE — The sample of anti-stripping agent shall be drawn within 24 h after delivery and tested within 7 days from the date of drawing, unless otherwise specified.

6.1.1 *Preparation of Samples*

Before carrying out any of the tests, the sample shall be mixed by gentle shaking to ensure uniformity.

6.2 If the single sample from a single run fails to fulfill the test requirements specified under **5**, sample shall be drawn on the basis of **6.1** for testing in the same manner. If these samples conform to requirement of 5, the lot shall be accepted otherwise the lot shall be rejected.

7 TESTS

Unless otherwise specified, tests shall be carried out according to the methods referred to in Table 1.

8 MARKING

8.1 Each container shall be legibly and indelibly marked with the following:

a) Name of the product;

b) Indication of the source of manufacture;

c) Month and year of manufacture; and

d) Batch number or code number.

8.2 BIS Certification Marking

The container may also be marked with the Standard Mark.

8.2.1 The use of the Standard Mark is governed by the provisions of *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufactures or producers may be obtained from the Bureau of Indian Standards.

9 APPLICATION

9.1 Anti-stripping agents can be used for the following two purposes:

a) For Bituminous Surfacing (Sprayed Work)

It covers road construction like surface dressing, penetration macadam, built up spray grout, etc.

b) For Bituminous Surfacing (Premixed Surfacing)

It covers bituminous surfacings like open graded premix carpet, bituminous macadam, semidense asphaltic concrete and dense asphaltic concrete.

9.2 Application of Anti-Stripping Agents for Bituminous Surfacing (Sprayed Work)

For this purpose the anti-stripping agent must be well mixed with the binder. The type of anti-stripping agent supplied is either liquid or powder. It will be fed into the bulk distributor through the manhole preferably fitted with a stirring device. The binder with agent shall be circulated by means of spray pump for at least 30 min to ensure homogeneous mixing. The additive can also be kept heated in a special storage tank attached to the bitumen tank. It shall be fed by a small dosage pump into the pipe supplying the bitumen throughout the operation. This ensures that the agent is mixed in the tank and also saves times.

In case the anti-stripping agent is to be added to the bitumen boiler, the agent in powder or liquid form shall be added to the boiler and agitated either manually or mechanically till thorough mixing of the anti-stripping agent is ensured. The stirring shall continue as the work progresses.

9.3 Application of Anti-Stripping Agents for Bituminous Surfacing (Premixed Surfacing)

In asphalt plant, the aggregate is normally dried and heated then mixed with bitumen. The mixing temperature varies between 140°C and 150°C for hot mixes, depending mainly on the grade of bitumen. With moist and partially wet aggregate the desired adhesion can be achieved by addition of anti-stripping agent. The amount of anti-stripping agent to be added depends upon the type and viscosity of the bitumen and type and gradation of aggregate. When very high viscosity bitumen is used and/or the aggregate gradation is dense,the dosage can be reduced.

Fatty acid amine dissolve easily in bitumen products if the bitumen temperature is 100-120°C. There are two methods for adding an agent. The most commonly used is to pour the calculated amount into the bitumen tank and allow 15-30 min of circulation to ensure a homogeneous mix and in this case it is necessary to use heat stable anti-stripping agent which can be stored in hot bitumen for 5-6 h without any significant loss in efficiency. In the other method the anti-stripping agent is injected into the bitumen just before it is sprayed on the aggregate in the mixer. With this system no mixing time in bitumen is required.

In the drum mix process, the aggregate is mixed with bitumen in the drum. Very often the aggregate contains a certain amount of moisture content normally remains in the aggregate after mixing. The required adhesion in the process can be ensured by use of an anti-stripping agent.

10 STORAGE OF ANTI-STRIPPING AGENTS

10.1 Anti-stripping agents are stable at normal temperature for many years provided they are stored in closed drums, barrels or tanks.

10.2 The anti-stripping agents are freeze thaw stable, but prolonged storage at low temperature may cause liquid anti-stripping agents to crystallize. These crystals should be dissolved on heating.

10.3 Solid anti-stripping agents should be stored at temperature below their melting point (40-50°C).

10.4 Steel or aluminium containers such as drums, tanks, etc, are suitable for storage of anti-stripping agents. Gaskets and hoses, made from Teflon or Polyamide should be used.

11 HANDLING AND SAFETY PRECAUTIONS

11.1 Anti-stripping agents cause irritation when in contact with skin and eyes. They are harmful to swallow and their vapours should not be inhaled. During prolonged skin contact anti-stripping agents are absorbed through skin and burns may occur.

11.2 When handling anti-stripping agents, always use protective gloves, protect eyes with goggles. Remove the contaminated clothing and protective devices as soon as possible and wash thoroughly before reuse (use 3 percent acetic acid). They should be kept away from public, specially from children.

11.3 In case of contact with skin, remove the excess anti-stripping agent, wash with diluted 3 percent acetic acid, and then with soap and water and finally rinse with plenty of water. If symptoms arise, apparently due to contact, seek medical attention immediately.

In case of contact with eyes, flush with 0.5 percent acetic acid and then irrigate with water for outlast 15 min. Seek medical attention.

11.4 In case of fire, quench with carbonic acid snow or chemical powder.

11.5 If spillage has occurred, absorb with earth, sand or sandiest and remove to a safe place. Solid amine may be swept up and filled in paper bags.

12 ADDITIVE DOSAGE

12.1 The dose of anti-stripping agent depends upon the nature of aggregates, that is, hydrophobic or hydrophilic and the percent voids in the mix. Higher quantity of dose is required for open surfaces like surface dressing bituminous macadam, open graded premix carpet and mix seal surfacings. In the case of dense bituminous mix a lesser quantity of anti-stripping agent is required.

12.2 The actual dose of anti-stripping agent shall also depend on the quality of anti-stripping agent. The stripping value of aggregate shall be determined as per procedure laid down in IS 6241. The method for determination of dose concentration is detailed in Annexes C and D.

ANNEX A

[*Table* 1, *Sl No.* (v)]

TEST FOR SOLUBILITY OF ANTI-STRIPPING AGENT IN HIGH SPEED DIESEL OIL

A-1 OUTLINE OF THE METHOD

The test determines the solubility of anti-stripping agent in bituminous binder. As bitumen is opaque (black colour material), high speed diesel oil of the same family is taken to determine the solubility of anti-stripping agent.

A-2 REAGENT

A-2.1 High Speed Diesel Oil

A-3 PROCEDURE

98 ml of the diesel oil and 2 ml of anti-stripping agent are taken in a measuring cylinder. The cylinder along with its contents is kept in water-bath maintained at 50 ± 1°C for half an hour. Take out the cylinder from water-bath and shake vigorously for 10 min. Remove the stopper, keep the cylinder in water-bath for 24 h and examine for separation/settlement.

A-4 REPORTING OF RESULTS

An anti-stripping agent is reported to be completely soluble in high speed diesel oil if no separation/ settlement is observed after 24 h.

ANNEX B

[*Table* 1, *Sl No.* (vi)]

TEST FOR STABILITY OF ANTI-STRIPPING AGENT

B-1 OUTLINE OF THE METHOD

This test determine whether any degradation in quality of anti-stripping agent is caused as a result of its storage in hot bitumen at 163°C for 5 h.

B-2 REAGENTS

B-2.1 Cut-back bitumen MC -3 (4 parts of bitumen and one part of kerosene.).

B-3 PROCEDURE

Blends of anti-stripping agent and bitumen to be used are prepared with 0.0, 0.25, 0.50, 0.75, 1.00, 1.25 and 1.50 percent content of anti-stripping agent and kept in an oven at 163°C for 5 h. After heat exposure, the blend samples are fluxed with kerosene oil to obtain cut-back bitumen of consistency of MC-3 and tested for under water coating test described in Annex C.

B-4 REPORTING OF TEST RESULTS

Anti-stripping agent is deemed to be heat resistant if the dose requirement before and after heat exposure remain the same.

ANNEX C

[*Clauses* 12.2 *and* B-3 and *Table* 1, *Sl No*. ix (a)]

UNDER WATER COATING TEST (FOR SPRAYED WORK)

C-1 GENERAL

This test is meant for dose determination of anti-stripping agent for sprayed work.

C-2 PROCEDURE

Anti-stripping agent shall be blended in 0.2, 0.4, 0.6, 0.8 and 1.0 percent in cut-back bitumen MC-3 (4 Parts of bitumen 80/100 and 1 percent of kerosene oil). These blends shall be used for testing their efficacy to coat the road aggregates under water.

In a tin container of 1-litre capacity, about 200 g of coarse aggregate of 13.2 mm nominal size (passing 22.4 mm sieve and retained in 11.2 mm sieve) shall be taken. The container shall be filled to 3/4th of its capacity with distilled water at 40°C. The bitumen anti-stripping agent blend shall be added at rate of 5.0 percent by weight of aggregate. The lid shall be replaced and container shall be vigorously shaken for two minutes. The water shall be drained off and aggregate piece shall be transferred on to a pieces of white paper and examined visually for coating. The test shall be repeated for different percentages of anti-stripping agent.

C-3 REPORT

The percent content of anti-stripping agent capable of giving 100 percent coating of coarse aggregate shall be taken as optimum dose for sprayed work.

ANNEX D

[*Clause* 12.2 and *Table* 1, *Sl No.* ix (b)]

COATING RETENTION TEST (FOR PREMIX WORK)

D-1 GENERAL

This test is meant for dose determination of anti-stripping agent for premix work.

D-2 PROCEDURE

The anti-stripping agent shall be blended in percent content 0.2, 0.4, 0.6, 0.8 and one percent in bitumen 80/100. These blends shall be used for testing their efficacy in retaining bitumen film on coated aggregates.

200 g of coarse aggregates of 13.2 mm nominal size (passing 22.4 mm sieve and retained on 11.2 mm sieve) shall be heated to 150°C and mixed with 5 percent blend of bitumen and anti-stripping agent, by weight of aggregates in a casserole with a spatula. After complete coating the mixture shall be transferred to a 500 ml beaker and allowed to cool at the room temperature for about two hours. Distilled water shall then be added to submerge the coated aggregates. The beaker shall be kept in a water-bath maintained at 40°C. Care is taken that the level of water in the water-bath is kept up to at least half the height of the beaker. After 24 h the beaker is taken out cooled to room temperature and percent area exposed is estimated visually while specimen is still under water. The test shall be repeated for different percentages of anti-stripping agent.

D-3 REPORT

The percentage of anti-stripping agent which retains 100 percent coating of the aggregate shall be taken as optimum dose for premix work.

ANNEX E

[*Table* 1, *Sl No.* (xi)]

DETERMINATION OF RETAINED MARSHALL STABILITY

E-1 OUTLINE OF THE METHOD

A numerical index of reduced marshall stability is obtained by comparing the marshall stability of freshly moulded and cured samples with the marshall stability of duplicate samples that have been immersed in water for 24 h.

E-2 APPARATUS

E-2.1 Water-Baths

E-2.1.1 Manually or automatically control water-bath for bringing the immersed test samples to 60 ± 1°C for marshall test.

E-2.1.2 *Water-Bath for Density Measurement*

An ordinary water-bath for weighing the test samples in water in order to determine their densities.

E-2.2 Balance — Suitable for weighing the test samples in air and water.

E-2.3 Transfer Plates — Sufficient number of flat glass plates of sufficient size to keep the test samples.

NOTE — The water used for the wet storage of test samples shall be either distilled or otherwise treated to eliminate electrolytes.

E-3 PROCEDURE

For quantitative evaluation, the test shall be carried out as Marshall method.

The Marshall test specimens shall be prepared using 5 percent bitumen 80/100 as well as bitumen blended with 1.0 percent anti-stripping agents (unexposed, heat exposed and air exposed). The test samples shall give a void content of about 6 percent. Atleast 6 Marshall specimens for each of the blend shall be prepared. Each set of 6 test specimens shall be sorted out in two groups of 3 each so that the average specific gravity of the specimens in Group I shall be almost the same as in Group II. Group I specimen shall be tested for Marshall stability as per the standard Marshall procedure, storing the specimen in water at 60°C for 30 to 40 min. Group II specimens shall be immersed in water at 60°C for 24 h and then tested for Marshall specimens shall be computed separately and retained Marshall stability will be calculated as under:

$$\text{Retained Marshall Stability, percent} = \frac{\text{Average Marshall Stability of Group II}}{\text{Average Marshall Stability of Group I}} \times 100$$

E-4 REPORT

The retained Marshall stability will be reported. A minimum retained Marshall stability of 90 percent has been stipulated for prevention of moisture damage in the field.

ANNEX F

(*Foreword*)

COMMITTEE COMPOSITION

Bitumen, Tar and their Products Sectional Committee, PCD 6

Organization	*Representative (s)*
Central Road Research Institute, New Delhi	Prof P. K. Sikdar (***Chairman***) Shri Sunil Bose (*Alternate* I) Dr P. K. Jain (*Alternate* II)
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Public Works Department, Mumbai	Shri V. B. Borge
Public Works Department, Tamilnadu	Chief Engineer

(*Continued on page* 8)

(*Continued from page 7*)

Organization	*Representative (s)*
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Synthetic Asphalts, Mumbai	Shri M. B. Jayawant
University of Roorkee, Roorkee	Prof H. C. Mehndiratta
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Member-Secretary
Shri T. Kalaivanan
Joint Director (PCD), BIS

Bitumen and Bituminous Products Subcommittee, PCD 6:2

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